

	DEPARTMENT OF COMMERCE National Institute of Standards and Technology National Voluntary Laboratory Accreditation Program	ISSUE DATE: February 19, 2004
	LAB BULLETIN	NUMBER: LB-10-2004
		LAP: Calibration
SUBJECT: Thermometry Proficiency Tests		

This bulletin applies to calibration laboratories accredited or seeking accreditation by NVLAP for-Resistance Thermometry. It provides the NVLAP Proficiency Test cutoff values for laboratories providing ITS-90 SPRT calibrations. The laboratory will be contacted by a NVLAP Program Manager to schedule the appropriate test(s). Laboratories that are providing these calibration services should refer to the following information developed by the Thermometry Group in the Process Measurements Division at NIST:

NVLAP Proficiency Test Cutoff Values for Laboratories Providing ITS-90 SPRT Calibrations

When laboratories are seeking or maintaining NVLAP accreditation for their ITS-90 SPRT calibration services with expanded uncertainties ($k=2$) less than or equal to the values given in Table 1, a need is indicated for a NIST Measurement Assurance Program (MAP) Test and the direct comparison of fixed-point cells. For those fixed-point cells that require a direct comparison with the NIST reference standards, a subset will be selected by the appropriate NIST Thermometry Group technical staff member. The use of the MAP and direct comparison of fixed-point cells as proficiency tests will be performed during alternate years. The MAP should be performed once every three years and the direct comparison of fixed-point cells once every five years. A single SPRT proficiency test covering the temperature range of the accreditation should be performed in the intervening years.

Table 1. MAP and direct comparison of fixed-point cell cutoff values. Cutoff values are for expanded uncertainties ($k=2$) in mK.

LN ₂	1.5	In FP	1.2
Ar TP	1.5	Sn FP	1.0
Hg TP	0.6	Zn FP	2.0
H ₂ O TP	0.3	Al FP	3.4
Ga MP	0.5	Ag FP	10.0

When laboratories are seeking or maintaining NVLAP accreditation for their ITS-90 SPRT calibration services with expanded uncertainties ($k=2$) within the range given in Table 2, a need is indicated for a NIST Measurement Assurance Program (MAP) Test. The use of the MAP as a proficiency test should be performed once every three years with a single SPRT proficiency test that covers the temperature range of the accreditation in the intervening years.

Table 2. MAP cutoff range. Cutoff ranges are for expanded uncertainties ($k=2$) in mK.

LN ₂	> 1.5 to 2.3	In FP	> 1.2 to 1.8
Ar TP	> 1.5 to 2.3	Sn FP	> 1.0 to 2.0
Hg TP	> 0.6 to 0.8	Zn FP	> 2.0 to 3.4
H ₂ O TP	> 0.3 to 0.5	Al FP	> 3.4 to 8.5
Ga MP	> 0.5 to 0.6	Ag FP	> 10.0 to 15.0

When laboratories are seeking or maintaining NVLAP accreditation for their ITS-90 SPRT calibration services with expanded uncertainties ($k=2$) greater than the values given in Table 3, a need is indicated for a single SPRT proficiency test that covers the temperature range of the accreditation. The full temperature range of accreditation should be tested once every three years. A single SPRT proficiency test covering a reduced temperature range of the accreditation should be performed in the intervening years.

Table 3. Single SPRT proficiency test cutoff values. Cutoff values are for expanded uncertainties ($k=2$) in mK.

LN ₂	> 2.3	In FP	> 1.8
Ar TP	> 2.3	Sn FP	> 2.0
Hg TP	> 0.8	Zn FP	> 3.4
H ₂ O TP	> 0.5	Al FP	> 8.5
Ga MP	> 0.6	Ag FP	> 15.0

The cutoff values given in Tables 1-3 are statistically derived from the ITS-90 SPRT calibration expanded uncertainties ($k=2$) claimed by NMIs from APMP, SIM, EUROMET, COOMET, and SADC MET.

The current cost of the proficiency tests as indicated in the above text is given in Appendix A (prices subject to change).

A list of additional information to have available for the on-site assessment is given in Appendix B.

A suggested list of uncertainty components used in assigning an uncertainty to an ITS-90 fixed-point cell is given in Appendix C.

Appendix A: FY2004 costs of the proficiency tests for those laboratories seeking or maintaining NVLAP accreditation for their ITS-90 SPRT calibration services (prices subject to change). Proficiency tests must be scheduled through NVLAP.

Proficiency Test	Temperature Range, °C	Cost, \$
MAP	-200 °C to 420 °C	14,858
MAP	-200 °C to 661 °C	16,511
Direct Comparison of a Fixed-Point Cell	to be determined with NIST staff	at cost
Single SPRT	-200 °C to 420 °C	4,092
Single SPRT	-200 °C to 661 °C	4,794

Other temperature ranges for each type of proficiency test are available upon request. Costs will be reflective of requested temperature range.

Appendix B: The following information should be available for the NVLAP on-site assessment of laboratories providing ITS-90 SPRT calibration services. Questions concerning the use of this list may be directed to Gregory Strouse, (301) 975-4803 or gstrouse@nist.gov.

This applies to each fixed point (Ar TP, Hg TP, H₂O TP, Ga MP or Ga TP, In FP, Sn FP, Zn FP, Al FP, and Ag FP).

Sample Purity: Show analysis of the effect of impurities on the realized temperature of the fixed-point cell (Type B, normal distribution). Use method 1 or 2, in combination with method 3: 1) total wt. % impurities, 2) total mole fraction impurities, and 3) derivation from analysis of experimental realization curves.

Phase Transition Repeatability: Using the check SPRT, measure multiple realizations ($n \geq 10$) of a fixed-point cell to calculate the phase transition repeatability value (Type A, standard deviation of the measurements). Supply a graph of the results.

Heat Flux: Quantify the heat flux by using the SPRT to measure the immersion profile of the phase transition realization of a fixed-point cell (Type B, normal distribution). For the SPRT to be near thermal equilibrium, the SPRT must be able to track the ITS-90 hydrostatic head effect over the bottommost 3 cm.

Phase Transition Realization Curves: Supply at least one freezing (where applicable) and melting curve.

The following should be provided for each measurement system used to measure the SPRTs.

Measurement System Repeatability: Supply a graph showing measurements of a thermostatically controlled reference resistor over at least a 3 h period to determine the repeatability of only the resistance ratio bridge. Supply a graph showing measurements of an SPRT in either a water or Ga fixed-point cell over at least a 3 h period to determine the repeatability of the measurement system under nominal SPRT calibration conditions.

Non-Linearity: Supply results giving the non-linearity of the resistance ratio bridge.

Calibration questions:

- 1) Prior to calibration, what is the stability requirement of the SPRT $R(\text{H}_2\text{O TP})$ during the annealing cycle?
- 2) What is the maximum allowable change of the $R(\text{H}_2\text{O TP})$ over the calibration of the SPRT?

Do you verify the calibration of the SPRT with measurements at redundant fixed-points (e.g., Ga MP or In FP)? If so, what are the acceptable limits of the non-uniqueness/calibration error [e.g., $W(\text{Ga MP})$ predicted from calibrated SPRT coefficients minus $W(\text{Ga MP})$ measured] assigned to the redundant fixed-point measurements?

Appendix C: A suggested list of uncertainty components used in assigning an uncertainty to an ITS-90 fixed-point cell follows. Questions concerning the use of this list may be directed to Gregory Strouse, (301) 975-4803 or gstrouse@nist.gov.

Bridge Repeatability
Bridge Resistance Ratio Measurement
Bridge Quadrature Effects (AC only)
Reference Resistor Stability
Phase Transition Realization Repeatability
Chemical Impurities
Hydrostatic Head Correction
SPRT Self-Heating Correction
Heat Flux
Gas Pressure
Slope of Plateau
Isotopic Variation (TPW only)
Propagation of TPW